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Diaquabis(picolinato *N*-oxide-κ^2O,O')-zinc(II)

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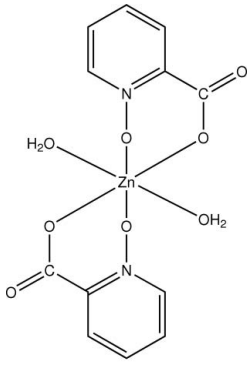
Key indicators: single-crystal X-ray study; T = 298 K; mean σ(C–C) = 0.007 Å; R factor = 0.046; wR factor = 0.149; data-to-parameter ratio = 11.0.

In the title compound, $[Zn(C_6H_4NO_3)_2(H_2O)_2]$, the Zn atom is located on a centre of inversion and shows a distorted octahedral coordination geometry. Two aqua ligands occupy the axial positions and four O atoms of the two chelating picolinic acid *N*-oxide ligands are located in the equatorial plane. Intermolecular hydrogen bonds between aqua ligands and organic ligands link molecules into a two-dimensional arrangement.

Related literature

For related literature, see: Bayot *et al.* (2006); Ciurtin *et al.* (2003); Lawrence *et al.* (1999); Meinrath *et al.* (2006); Shan *et al.* (2002); Steiner (2002); Yang *et al.* (2004); Zafar *et al.* (2000).



Experimental

Crystal data

$[Zn(C_6H_4NO_3)_2(H_2O)_2]$
M_r = 377.63
Monoclinic, $P2_1/c$
a = 6.6837 (5) Å
b = 15.7376 (13) Å
c = 6.9935 (6) Å
β = 115.3700 (10)°
V = 664.67 (9) Å^3
Z = 2
Mo $K\alpha$ radiation
μ = 1.90 mm^{-1}
T = 298 (2) K
0.21 × 0.18 × 0.16 mm

Data collection

Bruker SMART CCD diffractometer
Absorption correction: multi-scan *CrystalClear* (Rigaku, 2005)
T_{min} = 0.674, T_{max} = 0.733
3515 measured reflections
1170 independent reflections
1033 reflections with $I > 2\sigma(I)$
R_{int} = 0.019

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.046
$wR(F^2)$ = 0.149
S = 1.15
1170 reflections
106 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.56 e Å^{-3}
$\Delta\rho_{min}$ = −0.90 e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

Zn1—O3	2.049 (3)	Zn1—O1*W*	2.143 (3)
Zn1—O2	2.059 (3)		
O3—Zn1—O2	86.46 (11)	O2—Zn1—O1*W*^{i}	88.93 (12)
O3—Zn1—O1*W*^{i}	89.79 (11)		

Symmetry code: (i) $-x+1, -y+1, -z+1$.

Table 2
Hydrogen-bond geometry (Å, °).

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O1*W*—H1*WB*···O1^{ii}	0.90	1.98	2.753 (4)	143
O1*W*—H1*WA*···O1^{iii}	0.90	2.20	2.742 (4)	118

Symmetry codes: (ii) $-x, -y+1, -z+1$; (iii) $x+1, y, z+1$.

Data collection: *CrystalClear* (Rigaku, 2005); cell refinement: *CrystalClear*; data reduction: *CrystalClear*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *SHELXTL* (Sheldrick, 1999); software used to prepare material for publication: *SHELXTL*.

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: ER2040).

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supplementary materials

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Diaquabis(picolinato *N*-oxide-$\kappa^2 O,O'$)zinc(II)

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Comment

In the past decade, much attention has been paid to the design and synthesis of self-assembling systems with organic ligands containing N and O donors (Bayot *et al.*, 2006; Ciurtin *et al.*, 2003; Steiner, 2002; Zafar *et al.*, 2000). Picolinic acid N-oxide (PANO) is one such ligand and several crystal structures of complexes containing the PANO ligand have been reported (Yang *et al.*, 2004; Shan *et al.*, 2002; Lawrence *et al.*, 1999; Meinrath *et al.*, 2006). We report here the synthesis and crystal structure of the title complex, (I) (Fig. 1). In (I), the Zn atom is located on a crystallographic inversion centre and adopts a distorted octahedral coordination geometry. The coordination environment is defined by two pyridine N-oxide oxygen donors and two oxygen donors from the carboxylate groups located in the equatorial plane and two aqua O-atom donors located in the axial positions (Fig. 1). Selected bond lengths and angles are shown in Table 1. Intermolecular O1W—H1WA···O1, O1W—H1WB···O1 hydrogen bonds between water molecules and carboxylate groups connect the molecules of (I) into a two-dimensional network (Table 2 and Fig. 2).

Experimental

All chemicals were obtained from commercial sources and used without further purification. The title compound was prepared by the direct reaction of $Zn(OOCCH_3)_2.2H_2O$ (22.1 mg, 0.1 mmol) and picolinic acid N-oxide (13.9 mg, 0.1 mmol) in water solution. Colourless block-shaped single crystals were obtained by slow evaporation at room temperature for about three weeks.

Refinement

Positional parameters of all H atoms were calculated geometrically and were allowed to ride on the C atoms to which they are bonded, with $U_{iso}(H) = 1.2U_{eq}(C)$.

Figures

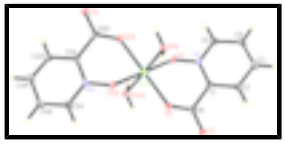


Fig. 1. The molecular structure of the title molecule, with the atom-numbering scheme. Displacement ellipsoids are drawn at the 30% probability level. Symmetry operation (A): [1 - *x*, 1 - *y*, 1 - *z*].

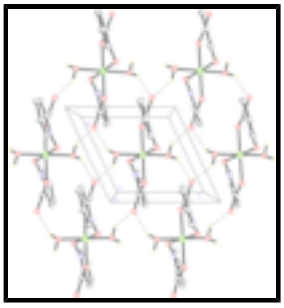


Fig. 2. A packing diagram of (I). Hydrogen bonds are shown as dashed lines.

Diaquabis(picolinato N-oxide-κ^2*O*,*O*')zinc(II)

Crystal data

$[Zn(C_6H_4NO_3)_2(H_2O)_2]$	F_{000} = 384
M_r = 377.63	D_x = 1.887 Mg m^{-3}
Monoclinic, $P2_1/c$	Mo $K\alpha$ radiation λ = 0.71073 Å
Hall symbol: -P 2ybc	Cell parameters from 25 reflections
a = 6.6837 (5) Å	θ = 7.5–15º
b = 15.7376 (13) Å	μ = 1.90 mm^{-1}
c = 6.9935 (6) Å	T = 298 (2) K
β = 115.3700 (10)º	Block, colourless
V = 664.67 (9) Å^3	0.21 × 0.18 × 0.16 mm
Z = 2	

Data collection

Bruker SMART CCD diffractometer	1170 independent reflections
Radiation source: fine-focus sealed tube	1033 reflections with $I > 2\sigma(I)$
Monochromator: graphite	R_{int} = 0.019
Detector resolution: 10 pixels mm^{-1}	θ_{max} = 25.0º
T = 298(2) K	θ_{min} = 2.6º
φ and ω scans	h = −7→7
Absorption correction: multi-scan CrystalClear (Rigaku, 2005)	k = −18→17
T_{min} = 0.674, T_{max} = 0.733	l = −6→8
3515 measured reflections	

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map
Least-squares matrix: full	Hydrogen site location: inferred from neighbouring sites
$R[F^2 > 2\sigma(F^2)]$ = 0.046	H-atom parameters constrained
$wR(F^2)$ = 0.149	$w = 1/[\sigma^2(F_o^2) + (0.0918P)^2 + 1.2258P]$

	where $P = (F_o^2 + 2F_c^2)/3$
$S = 1.15$	$(\Delta/\sigma)_{max} < 0.001$
1170 reflections	$\Delta\rho_{max} = 0.56$ e Å^{-3}
106 parameters	$\Delta\rho_{min} = -0.90$ e Å^{-3}
Primary atom site location: structure-invariant direct methods	Extinction correction: none

Special details

Geometry. All e.s.d.'s (except the e.s.d. in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell e.s.d.'s are taken into account individually in the estimation of e.s.d.'s in distances, angles and torsion angles; correlations between e.s.d.'s in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell e.s.d.'s is used for estimating e.s.d.'s involving l.s. planes.

Refinement. Refinement of F^2 against ALL reflections. The weighted *R*-factor *wR* and goodness of fit *S* are based on F^2, conventional *R*-factors *R* are based on *F*, with *F* set to zero for negative F^2. The threshold expression of $F^2 > \sigma(F^2)$ is used only for calculating *R*-factors(gt) *etc*. and is not relevant to the choice of reflections for refinement. *R*-factors based on F^2 are statistically about twice as large as those based on *F*, and *R*- factors based on ALL data will be even larger.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å^2)

	x	*y*	*z*	U_{iso}*/U_{eq}
Zn1	0.5000	0.5000	0.5000	0.0252 (3)
O1W	0.4917 (5)	0.43468 (18)	0.7653 (4)	0.0222 (7)
H1WA	0.6291	0.4170	0.8515	0.033*
H1WB	0.4423	0.4702	0.8365	0.033*
O1	−0.1745 (5)	0.49427 (16)	0.1316 (6)	0.0229 (7)
O2	0.1644 (5)	0.52224 (19)	0.3760 (5)	0.0218 (7)
O3	0.4215 (4)	0.38945 (17)	0.3288 (5)	0.0217 (7)
N1	0.2521 (5)	0.3424 (2)	0.3225 (5)	0.0168 (7)
C1	0.0530 (6)	0.3771 (2)	0.2833 (6)	0.0175 (8)
C2	−0.1198 (7)	0.3240 (3)	0.2650 (7)	0.0232 (9)
H2A	−0.2610	0.3483	0.2384	0.028*
C3	−0.0923 (8)	0.2365 (3)	0.2826 (7)	0.0287 (10)
H3A	−0.2135	0.1999	0.2667	0.034*
C4	0.1137 (8)	0.2034 (3)	0.3249 (7)	0.0273 (10)
H4A	0.1386	0.1432	0.3403	0.033*
C5	0.2825 (7)	0.2572 (3)	0.3436 (7)	0.0234 (9)
H5A	0.4259	0.2341	0.3737	0.028*
C6	0.0146 (6)	0.4725 (3)	0.2616 (6)	0.0177 (8)

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Zn1	0.0186 (5)	0.0236 (5)	0.0293 (5)	−0.0011 (2)	0.0064 (4)	−0.0029 (3)
O1W	0.0178 (14)	0.0239 (15)	0.0222 (14)	0.0006 (11)	0.0059 (11)	0.0006 (11)
O1	0.0128 (16)	0.0208 (16)	0.0256 (17)	0.0033 (10)	−0.0008 (13)	−0.0021 (11)
O2	0.0118 (14)	0.0176 (13)	0.0289 (16)	−0.0007 (11)	0.0020 (12)	−0.0062 (13)

O3	0.0131 (13)	0.0210 (14)	0.0313 (16)	−0.0047 (11)	0.0099 (12)	−0.0094 (12)
N1	0.0162 (16)	0.0150 (16)	0.0172 (16)	−0.0009 (12)	0.0051 (13)	−0.0036 (13)
C1	0.0165 (19)	0.0163 (19)	0.0172 (19)	0.0014 (15)	0.0048 (16)	−0.0027 (15)
C2	0.019 (2)	0.021 (2)	0.028 (2)	−0.0020 (16)	0.0087 (18)	−0.0012 (17)
C3	0.032 (3)	0.023 (2)	0.032 (2)	−0.0086 (18)	0.015 (2)	−0.0015 (18)
C4	0.039 (3)	0.014 (2)	0.029 (2)	0.0003 (17)	0.014 (2)	−0.0004 (17)
C5	0.027 (2)	0.0164 (19)	0.025 (2)	0.0044 (16)	0.0089 (19)	−0.0026 (16)
C6	0.0146 (19)	0.0186 (19)	0.022 (2)	0.0019 (16)	0.0101 (16)	0.0006 (16)

Geometric parameters (Å, °)

Zn1—O3^{i}	2.049 (3)	N1—C1	1.354 (5)
Zn1—O3	2.049 (3)	N1—C5	1.356 (5)
Zn1—O2^{i}	2.059 (3)	C1—C2	1.386 (6)
Zn1—O2	2.059 (3)	C1—C6	1.520 (5)
Zn1—O1W^{i}	2.143 (3)	C2—C3	1.389 (6)
Zn1—O1W	2.143 (3)	C2—H2A	0.9600
O1W—H1WA	0.9000	C3—C4	1.381 (7)
O1W—H1WB	0.9001	C3—H3A	0.9601
O1—C6	1.247 (5)	C4—C5	1.371 (6)
O2—C6	1.253 (5)	C4—H4A	0.9597
O3—N1	1.338 (4)	C5—H5A	0.9600
O3^{i}—Zn1—O3	180.00 (8)	O3—N1—C5	117.3 (3)
O3^{i}—Zn1—O2^{i}	86.46 (11)	C1—N1—C5	120.6 (3)
O3—Zn1—O2^{i}	93.54 (11)	N1—C1—C2	118.9 (4)
O3^{i}—Zn1—O2	93.54 (11)	N1—C1—C6	121.8 (3)
O3—Zn1—O2	86.46 (11)	C2—C1—C6	119.2 (3)
O2^{i}—Zn1—O2	180.0	C3—C2—C1	121.1 (4)
O3^{i}—Zn1—O1W^{i}	90.21 (11)	C3—C2—H2A	119.6
O3—Zn1—O1W^{i}	89.79 (11)	C1—C2—H2A	119.3
O2^{i}—Zn1—O1W^{i}	91.07 (12)	C2—C3—C4	118.3 (4)
O2—Zn1—O1W^{i}	88.93 (12)	C2—C3—H3A	120.8
O3^{i}—Zn1—O1W	89.79 (11)	C4—C3—H3A	120.9
O3—Zn1—O1W	90.21 (11)	C5—C4—C3	119.5 (4)
O2^{i}—Zn1—O1W	88.93 (12)	C5—C4—H4A	120.2
O2—Zn1—O1W	91.07 (12)	C3—C4—H4A	120.3
O1W^{i}—Zn1—O1W	180.00 (13)	N1—C5—C4	121.5 (4)
Zn1—O1W—H1WA	109.3	N1—C5—H5A	119.2
Zn1—O1W—H1WB	109.5	C4—C5—H5A	119.4
H1WA—O1W—H1WB	109.5	O2—C6—O1	125.2 (4)
C6—O2—Zn1	126.3 (3)	O2—C6—C1	119.9 (4)
N1—O3—Zn1	119.4 (2)	O1—C6—C1	114.8 (3)
O3—N1—C1	121.9 (3)		
O3^{i}—Zn1—O2—C6	−175.0 (3)	C2—C1—C2—C3	0(100)
O3—Zn1—O2—C6	5.0 (3)	C6—C1—C2—C3	179.7 (4)

O1W^{i}—Zn1—O2—C6	94.8 (3)	C1—C2—C3—C2	0(2)
O1W—Zn1—O2—C6	−85.2 (3)	C2—C2—C3—C4	0.0 (7)
O2^{i}—Zn1—O3—N1	137.7 (3)	C1—C2—C3—C4	1.8 (7)
O2—Zn1—O3—N1	−42.3 (3)	C2—C3—C4—C5	−1.4 (7)
O1W^{i}—Zn1—O3—N1	−131.2 (3)	C2—C3—C4—C5	−1.4 (7)
O1W—Zn1—O3—N1	48.8 (3)	O3—N1—C5—C4	−175.5 (4)
Zn1—O3—N1—C1	47.5 (4)	C1—N1—C5—C4	0.4 (6)
Zn1—O3—N1—C5	−136.6 (3)	C3—C4—C5—N1	0.3 (6)
O3—N1—C1—C2	175.7 (3)	Zn1—O2—C6—O1	−154.2 (3)
C5—N1—C1—C2	0.0 (6)	Zn1—O2—C6—C1	28.9 (5)
O3—N1—C1—C2	175.7 (3)	N1—C1—C6—O2	−36.9 (6)
C5—N1—C1—C2	0.0 (6)	C2—C1—C6—O2	142.3 (4)
O3—N1—C1—C6	−5.1 (5)	C2—C1—C6—O2	142.3 (4)
C5—N1—C1—C6	179.2 (4)	N1—C1—C6—O1	145.9 (4)
N1—C1—C2—C2	0.0 (2)	C2—C1—C6—O1	−34.9 (5)
C6—C1—C2—C2	0.00 (6)	C2—C1—C6—O1	−34.9 (5)
N1—C1—C2—C3	−1.1 (6)		

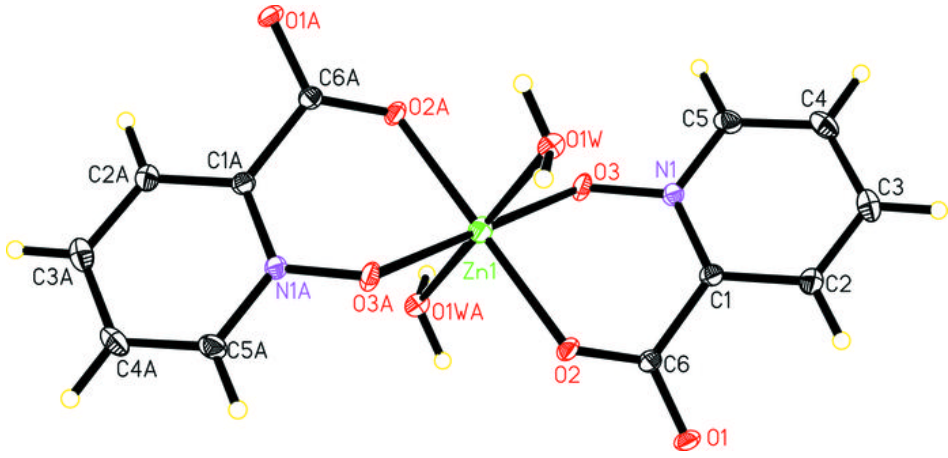
Symmetry codes: (i) −*x*+1, −*y*+1, −*z*+1.

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O1W—H1WB···O1^{ii}	0.90	1.98	2.753 (4)	143
O1W—H1WA···O1^{iii}	0.90	2.20	2.742 (4)	118

Symmetry codes: (ii) −*x*, −*y*+1, −*z*+1; (iii) *x*+1, *y*, *z*+1.

Fig. 1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C6A
O2A
C1A
C2A
C3A
N1A
O3A
C4A
C5A
Zn1
O1WA
O1W
O3
N1
C5
C4
C3
C2
C1
C6
O2
O1

Fig. 2